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The electronic structure of polymerized fullerenes and dimerized heterofullerenes

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Abstract. We present studies of the electronic structure of polymerized orthorhombic Rb_1C_{60} and dimerised $C_{59}N$ using electron energy-loss spectroscopy in transmission. From the C1s excitation spectra a reduced density of p^* states is observed for polymerized Rb_1C_{60}. This is in contrast to $(C_{59}N)_2$ and can be explained by the different type of 'doping' and by the different bonding between the fullerene molecules in the two systems. Additional information about the optical properties was obtained from the low energy loss function. Using a Kramers-Kronig analysis, the dielectric function, (e), and the optical conductivity, (s), have been derived. $e(0)$ and the onset of the spectral weight have been compared between the polymer, the dimer and C_{60}. This onset of spectral weight is found to be at 1.2 and 1.4 eV for o-Rb_1C_{60} and for $(C_{59}N)_2$, respectively.

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Polymeric fullerenes are of particular interest because of their possible applications as super-hard compounds [1]. In particular, the charged polymers of the A_1C_{60} (A=K, Rb, Cs) system have been widely investigated. The stable phase A_1C_{60} was first observed at elevated temperatures using Raman spectroscopy in the case of K_1C_{60} [2]. This $x = 1$ phase was found to crystallize at elevated temperature in an fcc lattice [3] and upon cooling to undergo either a first order phase transition into an orthorhombic o-A_1C_{60} phase for (A=Rb, Cs) [4], or to phase separate into pristine C_{60} and K_3C_{60}, as has been verified by photoemission (PES) [5], Raman and infrared (IR) spectroscopy [2, 6]. In addition, a metastable orthorhombic room temperature phase of K_1C_{60} was observed at high cooling rates [7]. The orthorhombic A_1C_{60} phases were found to be polymers, resulting from a [2 + 2] cycloaddilion process between the C_{60} molecules [8]. The polymer has orthorhombic crystal structure with point symmetry D_{2h} [13]. A_1C_{60} phases present the only air-stable alkali metal inlercalated compound [9] and in the case of K_1C_{60} show a quasi-one-dimensional metallic behaviour [4, 10]. The electronic structure of the o-Rb_1C_{60} phase has been analysed using PES [5, 11], electron energy-loss spectroscopy in reflection (HREELS) [11] and by studies of the C1s excitation edges in an electron microscope [12]. In the PES experiments, an additional structure below the prior HOMO of the carbon cage is observed which can be assigned to states from the partly filled molecular orbitals of the prior LUMO of the carbon cage (signaling charge transfer of the alkali metal s-electrons into the carbon-derived states). For the polymer phase, this lowest binding energy (BE) structure crosses the Fermi edge, suggesting a metallic phase. However, calculations of the polymerized o-A_1C_{60} compounds predict a finite energy gap [13], although this gap can be small - as low as 0.3 eV [14].

An additional new field in fullerene science is on-ball 'doping' by substituting a carbon atom by an atom of a different element (heteroatom). The synthesis and isolation of bulk quantities of the first so-called heterofullerene, $(C_{59}N)_2$ [15], have recently allowed the first analysis of the physical and chemical properties of these dimers in the solid state. The crystal structure of pristine $(C_{59}N)_2$ has been studied [16, 17] by x-ray diffraction on sublimed material (i.e. without remaining solvent) - confirming the dimerised nature of the heterofullerene which crystallises in a monoclinic structure and is thus, as regards the fullerene dimers, isostructural with the quenched Rb_1C_{60} dimer [18]. Recent calculations have also predicted that the structure of $(C_{59}N)_2$ is a dimer with only one C-C intermolecular bond and C_{2h} symmetry in contrast to the result of the [2 + 2] cycloaddition process in o-A_1C_{60}. The N atoms are in a *trans* configuration with a 'closed' C-N network on the ball [19]. This structure is also very similar to the molecular structure of isoelectronic dimersied C_{60}^- [20, 21]. Additional experimental evidence for this predicted configuration was found by analysing the parent hydroheterofullerene $C_{59}HN$ [22] and by using the N 1*s* excitation spectrum as a local probe of the chemical environment of the N site [23]. From a comparison between the valence band PES and the C 1*s* core level excitation spectra of the quenched monoclinic Rb_1C_{60} dimer and dimerised $C_{59}N$ the different nature of the 'doping' in these isoelectronic and isostructural compounds has been elucidated [23]. For the monoclinic Rb_1C_{60} dimer, similar to the o-Rb_1C_{60} polymer, a charge transfer of the Rb 5*s* electrons into the molecular orbitals of the fullerene molecule is observed, whereas the extra electron in the heterofullerene

remains strongly localized around the nitrogen site in full agreement with theory [23]. Therefore, $(C_{59}N)_2$ is the first example of a stable fullerene dimer. Interestingly, intercalation of $(C_{59}N)_2$ with potassium leads to a phase $K_6C_{59}N$, isostructural to K_6C_{60}, with the heterofullerene molecules separated in monomers [24].

In this contribution we report measurements of the loss function and the C 2*p* derived unoccupied electronic structure of the air-stable polymer o-Rb_1C_{60} probed by electron energy-loss spectroscopy (EELS) in transmission. We compared the results to those from pristine C_{60} and to the dimer of the heterofullerene $C_{59}N$. The significant differences in the electronic structure follow from the different kind of bonding in the dimer and the polymer and by the lower symmetry and different charge on the carbon cage compare to pristine C_{60}.

1 Experimental

For the o-Rb_1C_{60} samples, the surface of C_{60} single crystals (grown by a double temperature gradient technique [25]) was doped *in situ* in an IR-spectrometer as described elsewhere [9]. The undoped part of the single crystals was subsequently dissolved in toluene to give free-standing films of Rb_1C_{60}. The preparation of $(C_{59}N)_2$ has been described previously [15]. After purification by multi"-cycle high-performance liquid chromatography, solid $(C_{59}N)_2$ grains were obtained by evaporation of o-dichlorobenzene followed by recrystallization from a CS_2 solution. After degassing in ultra high vacuum (UHV) at 200°C for 48 h thin films of the $(C_{59}N)_2$ dimers were prepared by sublimation at 560°C onto KBr single crystals. In order to obtain free-standing films the KBr crystals were dissolved in distilled water.

In both cases a resulting film thickness of the fullerene of about 1000-1500 Å was chosen to minimize contributions from multiple scattering. The films were mounted on electron microscope grids and transferred into the purpose-built EELS spectrometer described elsewhere [26]. For the measurements, two different sets of energy and momentum solutions were chosen: 115 meV and 0.05 $Å^{-1}$ for the loss function in the low energy region, 160 meV and 0.1 $Å^{-1}$ for the C 1*s* core level excitation measurements. Electron diffraction studies both *in situ* and and in a transmission electron microscope gave diffraction patterns for o-Rb_1C_{60} and $(C_{59}N)_2$ consistent with the reported orthorhombic [4] and monoclinic [18] structures, respectively.

2 Results and Discussion

Fig. 1 shows a comparison of the C 1*s* excitation spectra of C_{60}, $(C_{59}N)_2$ and o-Rb_1C_{60}. The data are normalised to the intensity of the s^* onset at 290.5 eV. The core level excitation spectra are a measure of the matrix element weighted carbon 2*p* derived unoccupied DOS (neglecting the effects of the core hole). For both the polymer and the dimer, the C 1*s* excitation spectra are broader than that of C_{60}. For polymerized o-Rb_1C_{60} the strong broadening can be mainly attributed to the significant distortion of the fullerene cage following the [2 + 2] cycloaddition in the polymer which results in a strong splitting of the energy levels in these orthorhombic crystals (point group D_{2h}). In addition, a reduced intensity (≈ 5%

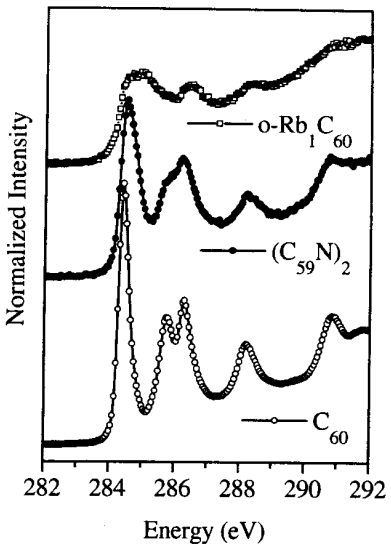

Fig. 1. C1*s* excitation spectra of C_{60} (o). o-Rb_1C_{60} (0), and $(C_{59}N)_2$ (•). The spectra are normalized to the edge at 290.5 eV and shifted in *y* direction

lower than in C_{60}) of the entire p^* derived states (≤ 290.5 eV) is observed. This cannot be solely explained in terms of the charge transfer of the Rb 5*s* electron into the LUMO of the carbon cage, but also signals the reduction in the p^* density caused by the formation of the four additional sp^3-hybridised carbon atoms per molecule at the connection points between the [2 + 2] polymerized C_{60} molecules. This is in contrast to the C 1*s* excitation spectrum of the uncharged $C_{59}N$ dimers. In this case only a relatively weak broadening but no reduction of the p^* density is observed within the experimental resolution. Moreover, for $(C_{59}N)_2$ not only the energy positions of the maxima but also the overall shape of the excitation spectra remains very similar compared to C_{60}. In fact, the spectral weight of all p^*-derived structures below the s^* edge at 292 eV is identical to that of C_{60} within the experimental error. This can be explained by a small distortion of the fullerene cage and therefore only a small splitting of the electronic states in full agreement with the reported monoclinic structure of the dimers with C_{2h} symmetry and with only one intermolecular C-C bond.

The loss function at low momentum transfer is related to interband transitions fulfilling dipole selection rules, thus giving a measure of dipole-allowed transitions (matrix element weighted excitations of the (s, p) electrons into empty (s^*, p^*) states) and after performing a Kramers-Kronig analysis (KKA) can be directly compared with optical measurements. In the upper panel of Fig. 2 we show the loss function up to 7 eV of the o-Rb_1C_{60} polymer (*solid line*) and C_{60} (*dotted line*) at a momentum transfer of 0.1 $Å^{-1}$. In the inset the loss function of the Rb_1C_{60} polymer is plotted in an extended energy region of 0 to 45 eV. The two strong maxima in the loss function at about 6.4 eV and about 23 eV (slightly lower than the 25 eV maximum for C_{60}) are characteristic for sp^2 carbon systems like graphite, conducting polymers, C_{60} or other fullerenes [26, 27, 29] and can be assigned to the so-called p plasmon (6.4 eV), reflecting the collective excitation of the p electrons, and the $p + s$ plasmon (23 eV), a collective excitation of all p and s derived valence electrons, respectively. For polymerized Rb_1C_{60} the $p \rightarrow p^*$ features be-

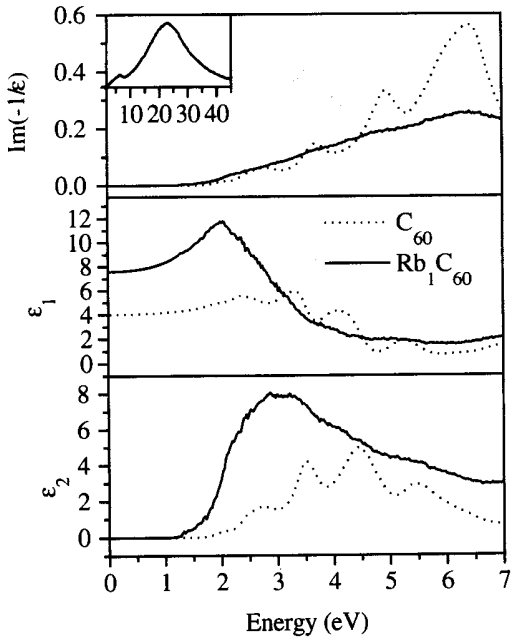

Fig. 2. Loss function (Im(-1/e)), real part (e_1) and imaginary part (e_2) of the dielectric function of o-Rb_1C_{60} (*solid line*) and C_{60} (*dotted line*)

tween 2 and 6 eV are significantly broadened compared to C_{60} and the maxima can barely be resolved in the loss function (only very broad and weak features at 2.2, 3.6, and 4.8 eV can be observed). Therefore, the lifting of the degeneracy of the distorted molecular energy levels due to the reduction from I_h to D_{2h} symmetry [13] and the charge transfer of the 5*s* electron from the Rb donor atom into the molecular orbitals of the carbon cage make it impossible to assign the individual optically allowed interband transitions in o-Rb_1C_{60}.

For semiconductors, the onset of the spectral weight is a direct measure of the optical gap. However, o-Rb_1C_{60} is a metal at room temperature, albeit a very poor one. The Drude contribution to the spectral weight should lead to a plasmon far below our energy resolution (the plasma edge is found at ≈ 0.1 eV [30]). This is in contrast to metallic K_3C_{60}, were the first plasmon is observed at 0.55 eV [31]. From optical reflectivity measurements this plasmon was found to consist of a small Drude part and a low lying interband transition [32]. This interband contribution can be explained by transitions between the three t_{1u} derived conduction bands. Calculations including intra- and interband transitions explaied the position, width and dispersion of this charge carrier plasmon in K_3C_{60} [33].

As we have shown that the free electron part is far below our energy resolution for o-Rb_1C_{60}, it is possible to calculate the dielectric function of the interband part of the transitions alone (all spectral weight above 0.4 eV) via a KKA of the loss function. The resulting real and imaginary part (e_1, e_2) of the dielectric function are depicted in the lower panels of Fig. 2. For the KKA the loss function was normalised using the oscillator strength sum rule:

$$n = N/V = \frac{m_e}{2\pi^2 e^2} \int_0^\infty E\,\varepsilon_2(E)\mathrm{d}E \qquad (1)$$

where E denotes the energy and m_e the mass of the electron, respectively. The number of valence electrons per Rb_1C_{60} unit is N = 248 (60 × 4 from carbon, 9 from Rb, one electron was subtracted to take into account the Drude contribution at low energy, which is not reachable in our measurements) and the volume of the primitive unit cell is V = 656.75 Å^3, as calculated from the measured lattice parameters [4].

From the loss function and the imaginary part of the dielectric function, we find an onset of the interband transitions ot about 1.2 eV which is, as already mentioned, well separated from the small Drude part observed in IR reflectivity measurements [30]. This onset of the spectral weight of the first interband transition is at significantly lower energy than that found for C_{60} (1.8 eV) [27], and it is in good agreement with the measured shoulder in UV/VIS spectra at 1.25 eV [28]. However, although the weak maxima in the loss function are found at the same position (2.2, 3.6 and 4.8 eV) our results are in contrast to the reported loss function of o-Rb_1C_{60} measured by HREELS [11] especially below 1.5 eV. In this work only a large asymmetrically broadened elastic peak was observed below 1.5 eV. This may arise from surface effects in the EELS in reflection experiment which are absent in our transmission experiments. As we observe no spectral weight below 1.2 eV (within the limit of our resolution) it is very unlikely that the Drude plasmon is located slightly below 0.3 eV as reported in [11]. In addition, from the KKA of the separated interband part of the optical transitions, the static background dielectric function at the low energy limit can be derived from the extrapolation of the real part of the dielectric function. This value $e_0 \approx 7.8$ reflects all contributions to the polarizability due to the dielectric background an is in very good agreement with the background dielectric function calculated from the reflectivity at 0.2 eV [9] (e(0.2 eV) = 7.2). The larger value of e_0 compared to C_{60} ($e_0 \approx 4$ [34]) also indicates a higher background polarizability and reflects the fact that the onset of the interband transitions is at lower energy for the polymer.

Additional information about the symmetry of the first interband transition can be obtained from measurements of the loss function at higher momentum transfer $\boldsymbol{q}$ (not shown). For C_{60} it has been confirmed by measuring the ($\boldsymbol{q}$) dependence of the loss function (at high $\boldsymbol{q}$ dipole allowed transition are suppressed in favour of monopole and quadrupole transitions) that the gap transition is a dipole forbidden excitonic $h_u \rightarrow t_{1u}$ transition at about ≈ 2 eV [29]. For the $\boldsymbol{q}$-dependence of the o-Rb_1C_{60} polymer, we find that the very broad features in the loss function at about 2.2, 3.6, and 4.8 eV decrease with increasing $\boldsymbol{q}$ and therefore are dipole allowed. No significant change with increasing $\boldsymbol{q}$ is found for the spectral onset at 1.2 eV. This may be explained by close lying dipole allowed and forbidden transitions that cannot be resolved. This behaviour is very similar to that of dimerised $C_{59}N$ as has been reported previously [35].

In Fig. 3 the real part of the optical conductivity (s_1) is plotted for o-Rb_1C_{60} (*solid line*), $(C_{59}N)_2$ (*dashed line*) and C_{60} (*dotted line*). The optical properties of dimerised $C_{59}N$ have been derived from the calculated dielectric function as described elsewhere [35]. For C_{60}, maxima of the spectral weight are observed at about 2.8, 3.5, 4.5, and 5.6 eV. In the case of o-Rb_1C_{60}, the structures in s_1 are observed at about 3.5, 4, 4.5, and 5.5 eV. In addition, a broad shoulder at 2.7 eV is visible, which can be related to the 2.8 eV peak of the pristine compound. In addition we find that compared to

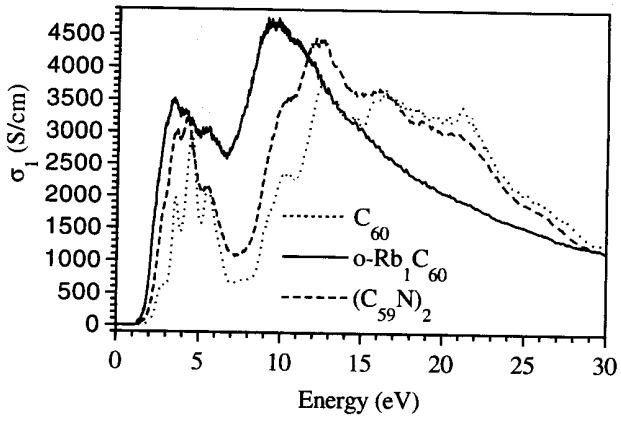

Fig. 3. Real part of the optical conductivity (s_1) of C_{60} (*dotted line*), dimerised $C_{59}N$ (*dashed line*), and o-Rb_1C_{60} (*solid line*)

C_{60} and dimerised $C_{59}N$ the maximum of the spectral weight of the optical transitions from and into *s* derived states is downshifted by about 3 eV and therefore is partly overlapping with the $p \rightarrow p^*$ transitions. The peaks in s_1 are located at3.5, 4, and 5.6 eV and an additional shoulder is found at 2.7 eV. From the onset of the spectral weight for dimerised $C_{59}N$ an optical gap of 1.4 eV, 0.4 eV lower than in C_{60}, is found. This downshift of 0.4 eV is also found for the change of the HOMO→LUMO gap in the calculated Kohn-Sham energy level diagram [19].

3 Conclusion

A strong broadening of the carbon 2*p*-derived unoccupied DOS and a reduction in the total number of *p** derived states has been observed for polymerized o-Rb_1C_{60} in comparison with C_{60}. This cannot be solely explained by the distortion of the carbon cage and the charge transfer of the Rb 5*s* electron into the molecular orbitals of the carbon cage. The *p** density is additionally reduced by the four sp^3-hybridised carbon atoms at the connection points between the C_{60} molecules. This is in contrast to the $C_{59}N$ dimers, where only a rather weak broadening due to the small distortion of the cage, but no reduction of the *p** density is observed within the experimental error.The energy loss spectra for low momentum transfer is also less structured for the polymer and the dimer compared to C_{60}. In both materials the onset of spectral weight is found at lower energy than that in C_{60}. The oberserved onsets in the spectra at 1.2 and 1.4 eV for o-Rb_1C_{60} and for $(C_{59}N)_2$, respectively, are in good agreement with optical measurements and calculations. Additionally in contrast to [11], no structure either from a Drude plasmon or from an interband transition is observed for the o-Rb_1C_{60} polymer between 0.4 and 1.2 eV, as it is also expected from the IR-reflectivity measurements and optical absorption measurements.

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